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INVESTIGATIONS LEADING TO THE
DESIGN AND DEVELOPMENT OF
MAGNESIUM/MAGNESIUM PERCHLORATE BATTERIES

OCTOBER 1, 1964 through DECEMBER 31, 1964

REPORT NO. 2

SECOND QUARTERLY PROGRESS REPORT

CONTRACT NO. DA-28-043-AMC-00221(E)

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CONTRACT NO. DA-28-043-AMC-00221(E)

DEPARTMENT OF THE ARMY PROJECT NO. 1G622001 A0530200

USAEL TECHNICAL GUIDELINES
EPP NO. 58274
DATED JUNE 20, 1964

U. S. ARMY ELECTRONICS LABORATORIES
POWER SOURCES DIVISION, ELECTRONICS COMPONENTS DEPT.
FORT MONMOUTH, NEW JERSEY

THE EAGLE-PICHER COMPANY
COUPLES DEPARTMENT
P. O. BOX 47
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J. M. Dines, Engineering Manager

DATE OF REPORT: January 31, 1965

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FIGURES

I. PURPOSE

The purpose of this work is to conduct investigations which will lead to the development of reliable, production-feasible magnesium/magnesium perchlorate batteries; to carry on research and development on the use of magnesium perchlorate as an electrolyte; toward determining the applicability of the system in military field use.

The investigations should provide practical design information which can be used for future battery development.

II. ABSTRACT

This abstract describes the significant accomplishments made on the research and development of magnesium/magnesium perchlorate batteries during the past three months.

Efficiency has been improved by the addition of lithium chromate to the magnesium perchlorate. Studies have indicated that the addition of one gram of lithium chromate per 100 ml of electrolyte will increase the anode efficiency in the range of five to fifteen percent.

Conductivity studies were carried out and studies indicated that four normal magnesium perchlorate is the most applicable to use. Data has also indicated the higher the normality of electrolyte, the lower the resistance.

Effort has been directed during the quarter to perfect a magnesium/magnesium perchlorate battery which would meet the requirements of the AN/PRC-62 specification, when discharged over a temperature range of -40°F to +125°F. The program was directed toward attaining the optimum performance at -40°F with the minimum number of cells per battery. After this was completed, two units were constructed and discharged at +70°F and +125°F.

Research in relation to positive and negative material was continued to reduce the cost of the battery and to keep its efficiency and reliability.

The AN/PRC-25 application was studied. The same problems that existed with the AN/PRC-62 application were found to exist in regard to this application.

Toward the end of the quarter, research and development was launched to meet the the specifications of the PPS-5 application.

III. REFERENCES AND CONFERENCES

A conference was held at Fort Monmouth on 27 October 1964. USAEL was represented by Mr. N. T. Wilburn, Dr. A. Fischbach, and others. Eagle-Picher was represented by Messrs. E. M. Morse and E. B. Cupp. A discussion of the efforts to date and future efforts directed toward the three specific applications occurred. It was decided that -40°F was a more practical temperature for the low temperature operation than the -65°F previously specified. In general, it was felt that the project was progressing satisfactorily at this time.

IV. INTRODUCTION

The general purpose and objectives of the present contract are fully outlined under the "Purpose" section of this report.

Although the original purpose and objectives permit a wide selection of basic components such as cathode materials, electrolyte solutes and anodes, the requirements and problems solved during this quarter of the contract have not necessitated utilizing all the possibilities. However, efforts have been carried out in a preliminary way to screen potential components to make certain the most useful would be the subject of detailed examination. The overall effort was planned and was generally followed along the outline below:

(1) equipment and techniques,

(2) electrolyte study,

(3) negative corrosion,

(4) overall battery test.

There may be variations within the report of this outline; however, this is an overall general outline which the report attempts to follow.

V. FACTUAL DATA AND DISCUSSION

A. Equipment and Techniques

Efforts directed toward establishing production techniques for producing batteries for the three field applications have consisted of refinements in the method of preparing the positive plates.

The vacuum-pasting process as described in the First Quarterly Report was modified during this work period. The process as described in the previous report consisted of depositing a batch of active material onto a filter paper, removing the excess water by vacuum and then pressing the material onto a grid. The modified process consisted of a continuous feed of a dense active material slurry onto the grid from a hopper. The thickness of the material is controlled by a gauged cutoff and the speed at which the grid moves under the hopper feed. Upon moving by the hopper, the pasted grid moves over a vacuum table where the excess moisture is removed. The continuous strip then passes through a set of rolls for pressing and sizing and then into a drying oven.

The equipment for a continuous operation of this sort has not been acquired. The operation has been used, however, utilizing equipment on hand and moving the materials by hand.

Plates manufactured by this method have been tested and found to compare very favorably with those produced by the batch process described in the previous report.

B. Cell and Battery Construction

There have been no notable changes in the cell construction techniques.

1. Battery Construction

a. Anodes

The anodes used consisted of AZ31B magnesium 0.014 inch thick with an apparent density of 28.6 grams per cubic inch.

b. Cathodes

Major emphasis during the quarter was placed on battery refinement; therefore, it was apparent that a larger batch of cathodic material was desirable. The blend size was increased from 300 grams to 1200 grams per batch. This material was blended by milling for eight hours in an 8.5 inch diameter ball mill using 60 porcelain balls. The cathodes were then fabricated using the vacuum deposition technique and placed on 4/0 expanded, .005 inch thickness silver grid at 1.35 grams per square inch. Following this operation, the plaques were pressed to an apparent density of 50-55 grams per cubic inch and dried. This material appeared to be uniformly blended and pasted excellent, requiring approximately 90 seconds for pasting each plaque. The A-4 formula used in the construction of these units was as follows:

Mercuric Oxide (Yellow)	84%
Carbon	10%
Divalent Silver-Oxide	4%
Carboxymethylcellulose.	1.5%
Solka-Flox	0.5%

c. Separation

The cell separator used in all units was 0.004 inch thickness filter paper. This material has proven reliable for this electrochemical system.

d. Cell Cases and Electrolyte Reservoir

Cell groups were assembled in the Y-4 cell case with the following inside dimensions:

Height	2.781 inches
Width	1.812 inches
Length	0.656 inch

As a reservoir for storage of the magnesium perchlorate, the above cell case height was reduced to 1.5 inches. This case was cemented as a cover to the standard cell case. As it became apparent that a 5.5 normal electrolyte concentration was not essential, the reservoir height was decreased to 1.187 inches which yielded an overall cell height of 4.62 inches, including the vent plug.

e. Terminals and Intercell Connectors

To conserve weight and to decrease the battery cost, threaded terminals were deleted in the construction of batteries MAP-2014-3 through 7. Silver wire lugs were countersunk into the cell case with a heating iron. The seal was secured by a coating of epoxy cement. A piece of .040 inch diameter silver wire was used to replace the heavier connector bar used in the construction of the first units.

The standard cell described above has been found to meet the requirements of both the AN/PRC-62 and AN/PRC-25 applications. Efforts to meet the PPS-5 application have required a larger (120 ampere-hour) cell design. The cell construction for this application has been scaled up from the smaller cells with no real changes tin techniques.

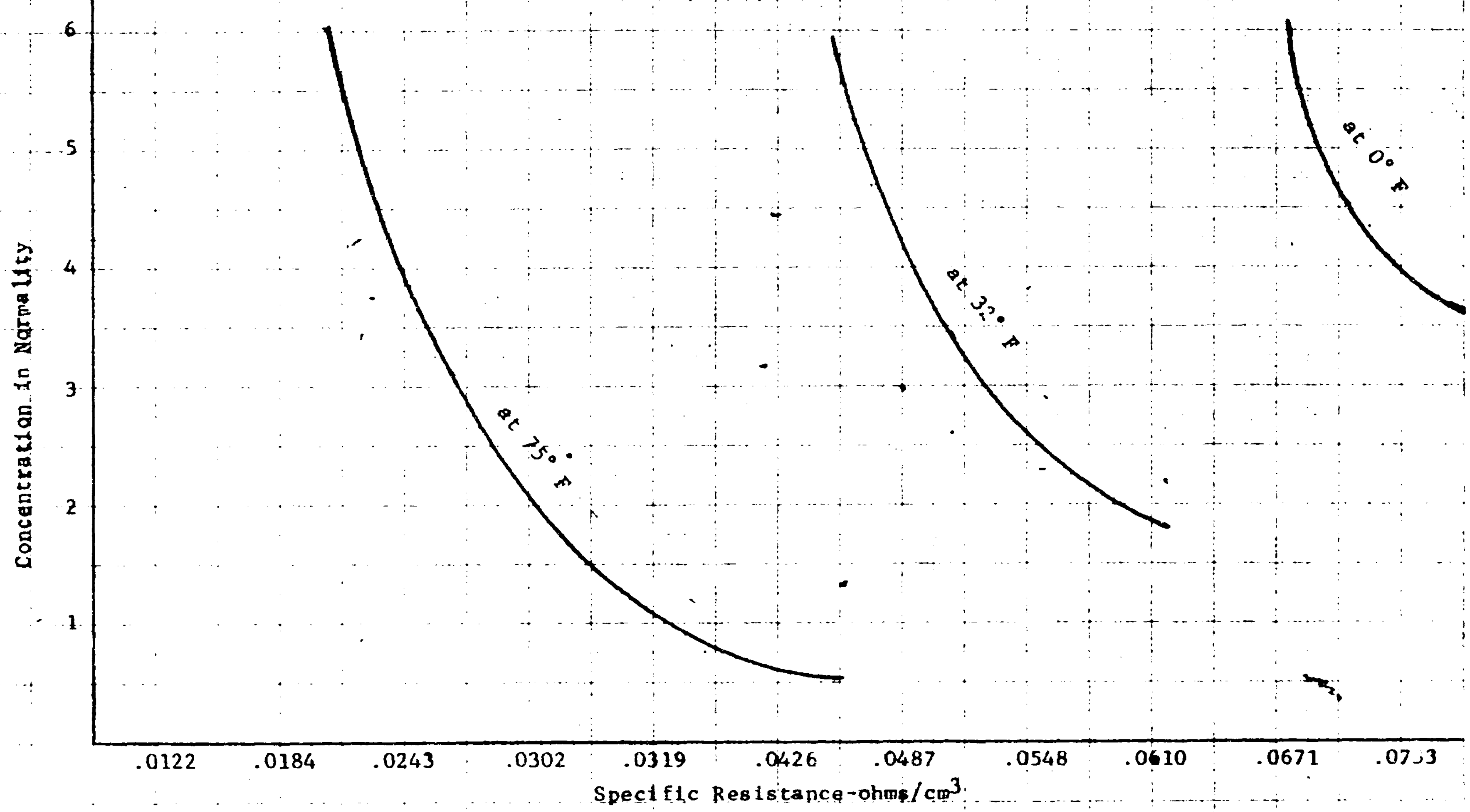
C. Electrolyte

Early cells tested at -65°F reported in the First Quarterly Report had indicated the desirability of using 5.5 to 6.0 normal Mg $(ClO_4)_2$ solution as electrolyte in the HgO/Mg system. Studies conducted during this quarter between the temperatures of -40°F and +125°F, however, have indicated 4.0 normal electrolyte to be adequate or even desirable.

During the past work period, a comparison of the electrical resistance of various concentrations of $Mg(ClO_4)_2$ in water were made. These measurements were made by immersing two simple sheet nickel electrodes into the solutions and passing 10,000 cycle A.C. current through the solution between the electrodes, and measuring the voltage drop across the electrodes. The results of these measurements are shown in Figure No. 8.

An analysis of the data from the freezing point curve and the heat of reaction indicated that concentrations lower than 4 normal might be used if the

FIGURE NO. 8
SPECIFIC RESISTANCE
AT VARIABLE TEMPERATURES



batteries were insulated to prevent heat loss at -40°F. Lower electrolyte normalities are desirable from the standpoint of lower cost, weight and total battery volume. However, the results of battery tests at -40°F comparing 4 N, 3 N, and 2 N electrolyte indicated that more is lost than gained by reducing the electrolyte concentration below 4 N. The results of these battery tests are shown in Figure Nos. 16 and 17 and are described in detail in the test section of this report.

D. Anode Corrosion Study

Efforts to extend the activated storage time, reduce the gassing rate and acquire good performance under low rate, long life operation, necessitated a study directed toward reducing the anode corrosion rate. Certain magnesium alloys were eliminated from further study during the past quarter due to their rapid rate of corrosion (Quarterly Report No. 1).

It was determined that the best possible alloy for use in this system was either AZ-21 or AZ-31. The difficulty in procuring AZ-21 in the size and thickness required has prohibited its use; therefore, the studies reported herein compare the characteristics of AZ-31 magnesium with pure sheet magnesium.

A study was initiated to measure the rate of no-load corrosion of AZ-31 alloy, the material selected for use, as compared to pure magnesium. The effects of certain additives to the electrolyte upon corrosion rate were also measured.

The test method consisted of collecting the gas displaced as the magnesium was oxidized and measuring milliters of gas per unit time.

The test samples consisted of a strip of the metal tested (1-9/16 in. x 2-1/4 in. x .015 in) in 4 normal electrolyte at 80°F. The pure magnesium caused the evolution of hydrogen at the rate of 5.5 ml/min. as compared to 0.383 ml/min. for the AZ-31 alloy. The rate of gas evolution for AZ-31 alloy is a cumulative figure. The initial gassing rate is quite rapid, the rate gradually slowing as the surface of the alloy becomes coated and passivates. The gassing of pure magnesium appears to be continuous.

Several additives to the electrolyte were spot-tested as corrosion inhibitors. These were:

Li_2CrO_4 $CaCrO_4$ Na_2CrO_4

$K_2Cr_2O_7$ $Na_2Cr_2O_7$ K_2CrO_4

Of these additives, Li_2CrO_4 was found to be the most effective in reducing magnesium corrosion.

As in the previous tests the oxidation rate was measured as a function of the H_2 displaced by the magnesium. After 72 hours of test enough pure magnesium had gone into solution to liberate 20 cc of gas at standard conditions giving a gassing rate of approximately 0.4 cc of gas per hour. The AZ-31 magnesium had displaced only 3.5 cc of H_2 over a 41-hour period, or 0.085 cc of H_2 per hour. One gram of magnesium is equivalent to 1,860 cc of H_2; therefore, at the rate of corrosion measured, nearly 22,000 hours would be required to dissolve 1 gram of AZ-31 alloy in the inhibited electrolyte.

Test cells were built up for the An/PRC-62 application and discharged utilizing the Li_2CrO_4 as an inhibitor. Figure Nos. 9 and 10 indicate that the Li_2CrO_4 lengthened the discharge time considerably.

FIGURE NO. 9
VOLTAGE-TIME
$HgO-Mg-Mg(ClO_4)_2$

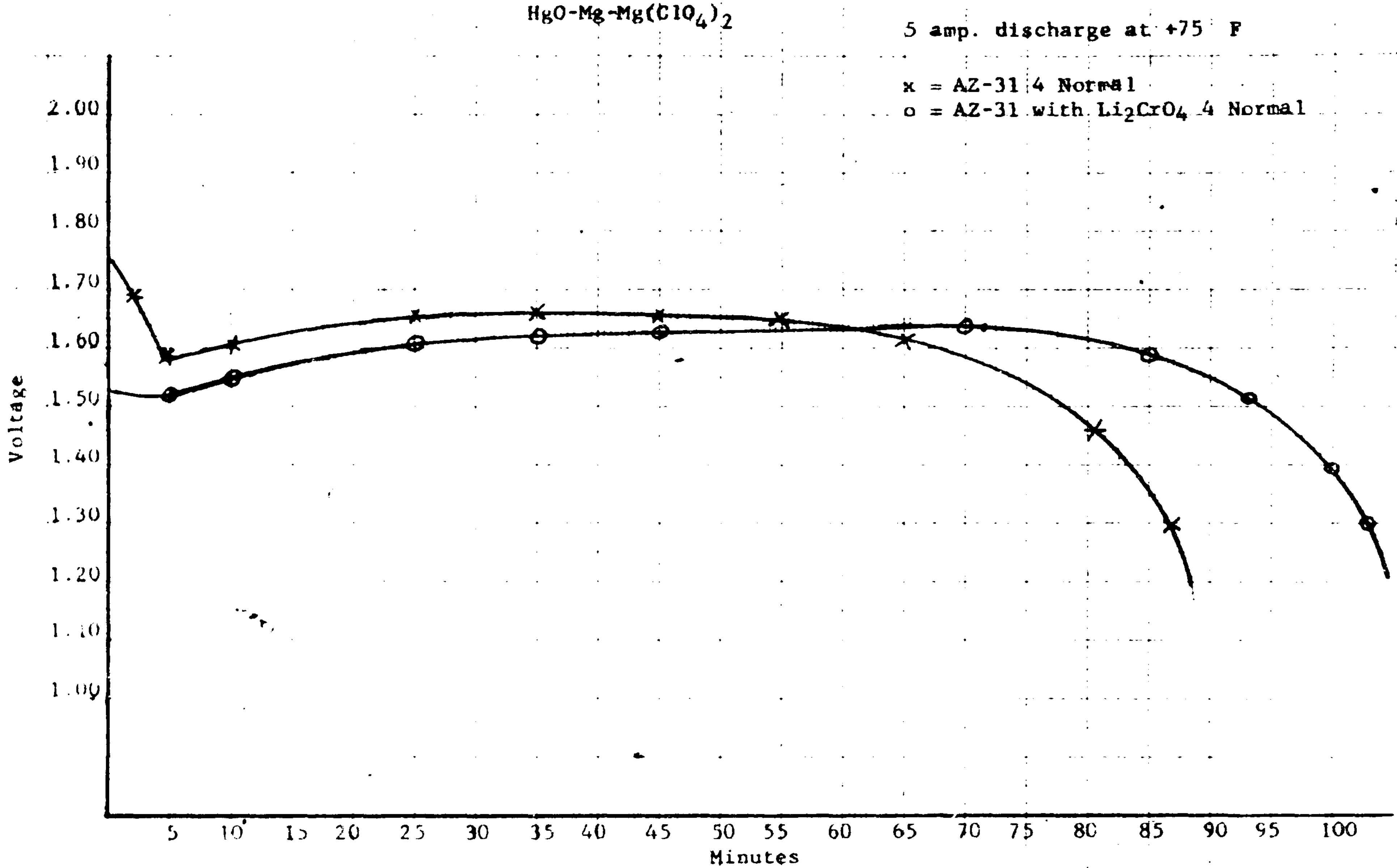
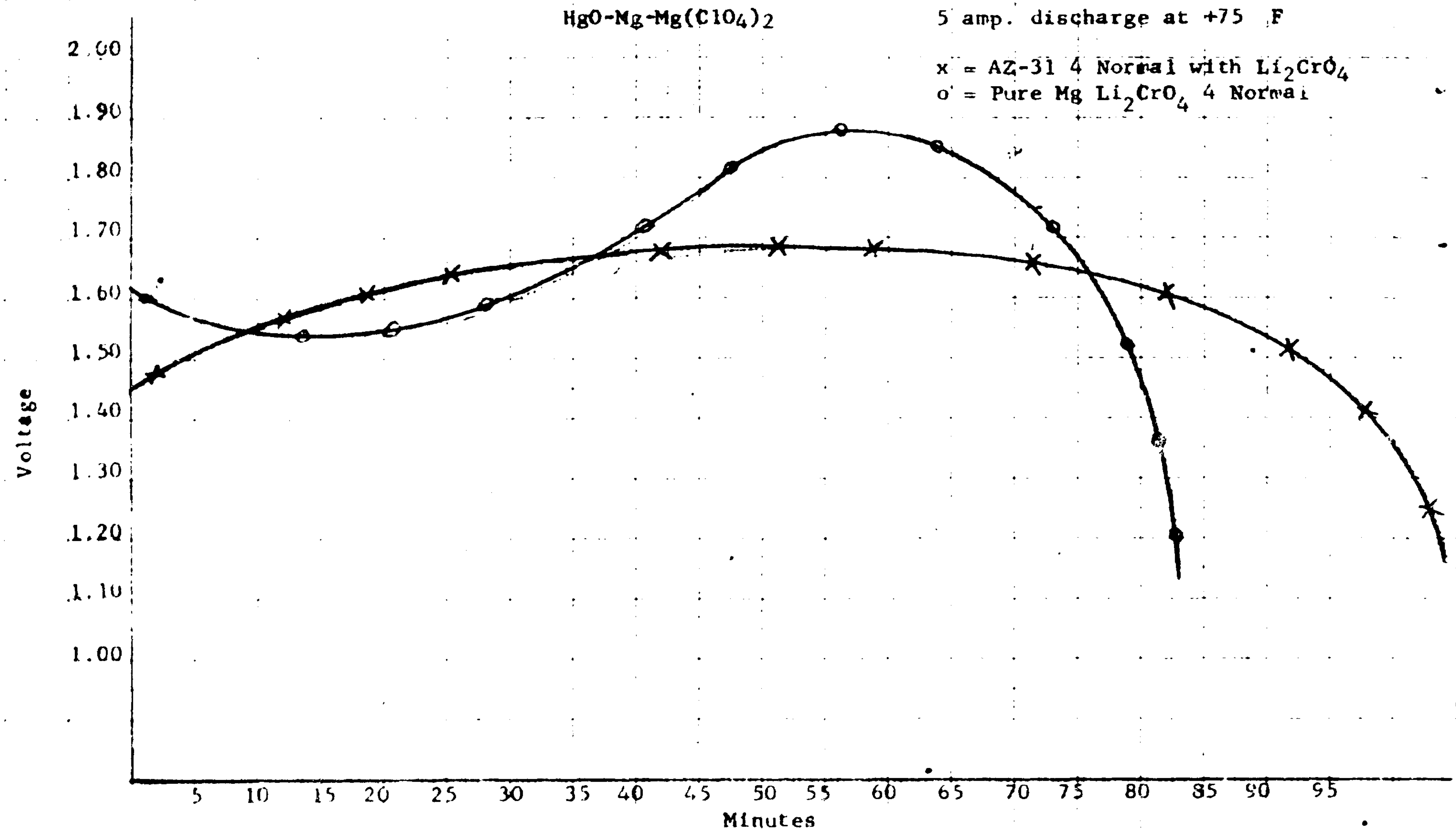


FIGURE NO. 10
VOLTAGE-TIME
$HgO-Mg-Mg(ClO_4)_2$



E. Cathodes

1. Grid Study

During the quarter, an attempt was made to reduce the cost of the battery. It was decided that cutting down on the cost of the grid wire would slice the cost of the battery considerably.

Six different types of grid wire were considered and tested:

1. 4/0 ex. Ni Grid
2. 2/0 ex. Ag Grid
3. 4/0 ex. Ag Grid
4. 4/0 ex. Ni Ag flashed
5. 4/0 ex. Cu
6. 4/0 ex. Cu Ag flashed

Data in Figure Nos. 11 and 12 indicate that 4/0 mesh Ag is the best for high voltage and performance. However, due to the high cost of silver, a cheaper material was needed that would maintain voltage and efficiency. Expanded 4/0 nickel was tested and found to have a wide voltage spread during the initial portion of discharge. Silver plated 4/0 nickel was tested. Silver plated 4/0 nickel maintained a lower voltage than the 4/0 silver and higher voltage than 4/0 nickel. It was decided that 4/0 nickel, silver plated, would be utilized in the AN/PRC-62 application due to the lower cost of the nickel, silver plating included, as compared to the high cost of silver.

Data in Figure Nos. 11 and 12 reveal a comparative study of the six different types of grid wire. Data indicates that the voltage and efficiency shown by cells utilizing the 2/0 silver, nickel and copper grids are not sufficient to meet the AN/PRC-62 application. However, since silver plated 4/0 nickel grid is economical and meets the specifications of the AN/PRC-62 application, it has been chosen as the desired grid material (4/0 Ni - $1.25/sq. ft, 2/0 Ag - $2.68/sq. ft, 4/0 Ag $3.10/sq. ft.).

FIGURE NO. 11

GRID WIRE STUDY

Voltage at 70 watt portion of AN/PRC-62 load profile
+75° F

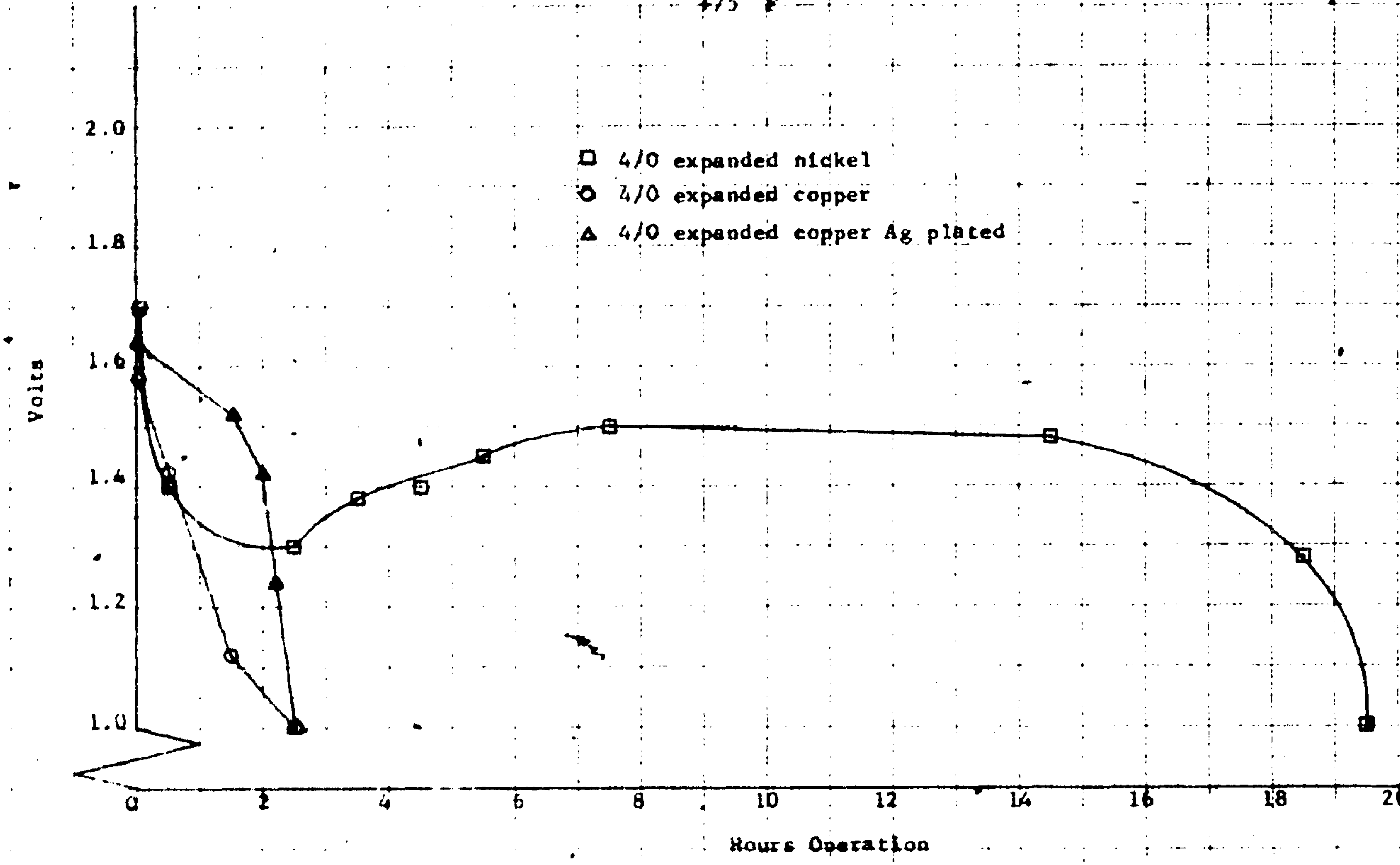
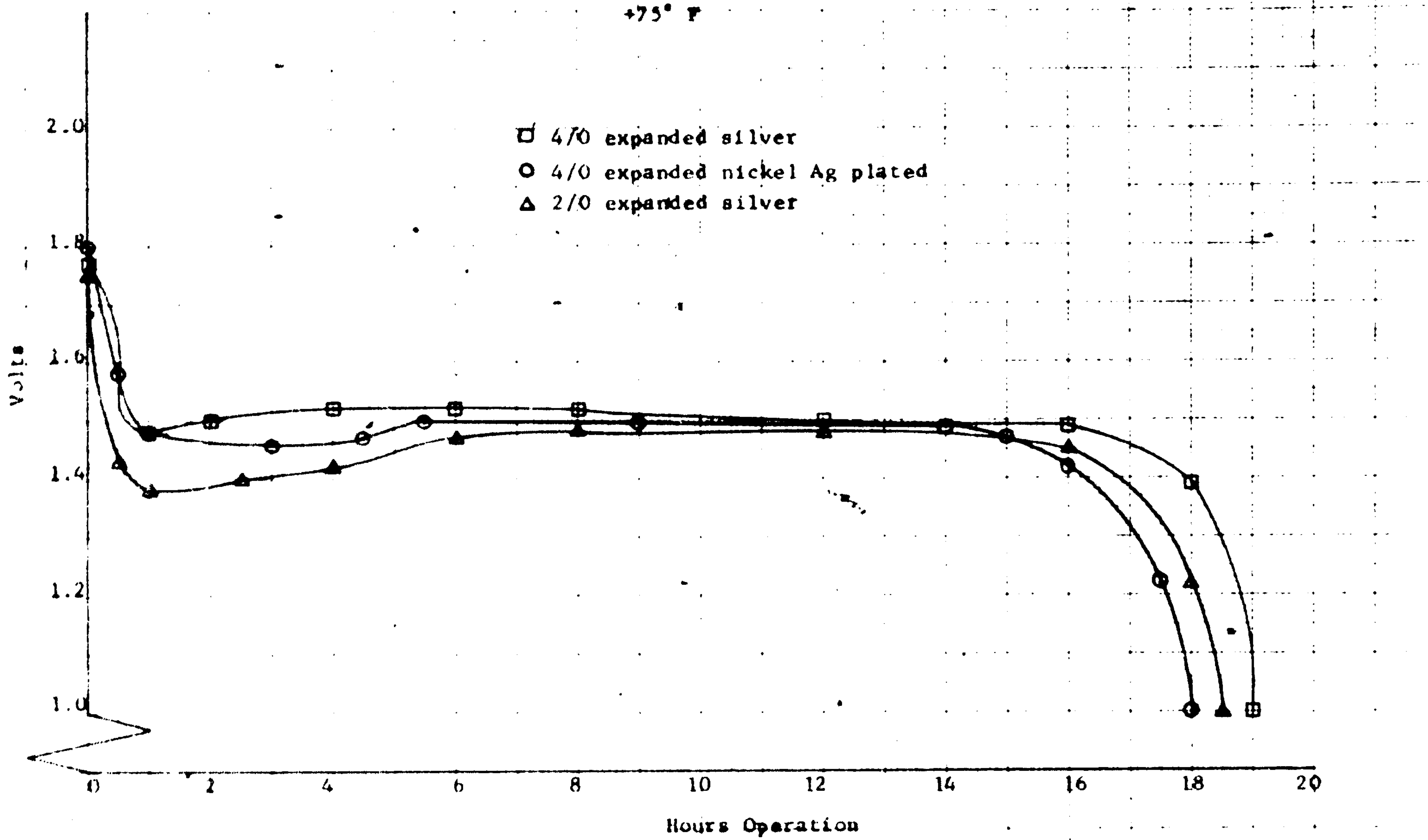


FIGURE NO. 12

GRID WIRE STUDY

Voltage at 70 watt portion of AN/PRC-62 load profile
+75° F



F. Battery Testing

1. Eleven Cell Batteries - (-40°F Discharge)

One eleven cell unit, S/N MAP-2014-2, was constructed with a sufficient amount of magnesium perchlorate to yield a 4.0 normal electrolyte. This unit was conditioned at -40°F for 16 hours, activated with +40°F water and discharged at the AN/PRC-62 specification rate. During discharge the battery was insulated by a polystyrene case with a wall thickness of 0.5 to 0.75 inch. As reported in the First Quarterly Report and revealed by Figure No. 13, this unit failed at the high rate surge after 20.75 hours of discharge yielding 91.4% positive efficiency at 8.91 ampere-hours, or 41 watt-hours per pound. Figure Nos. 14 and 15 display photographs of the battery prior to and after discharge.

2. Twelve Cell Batteries - (-40°F Discharge)

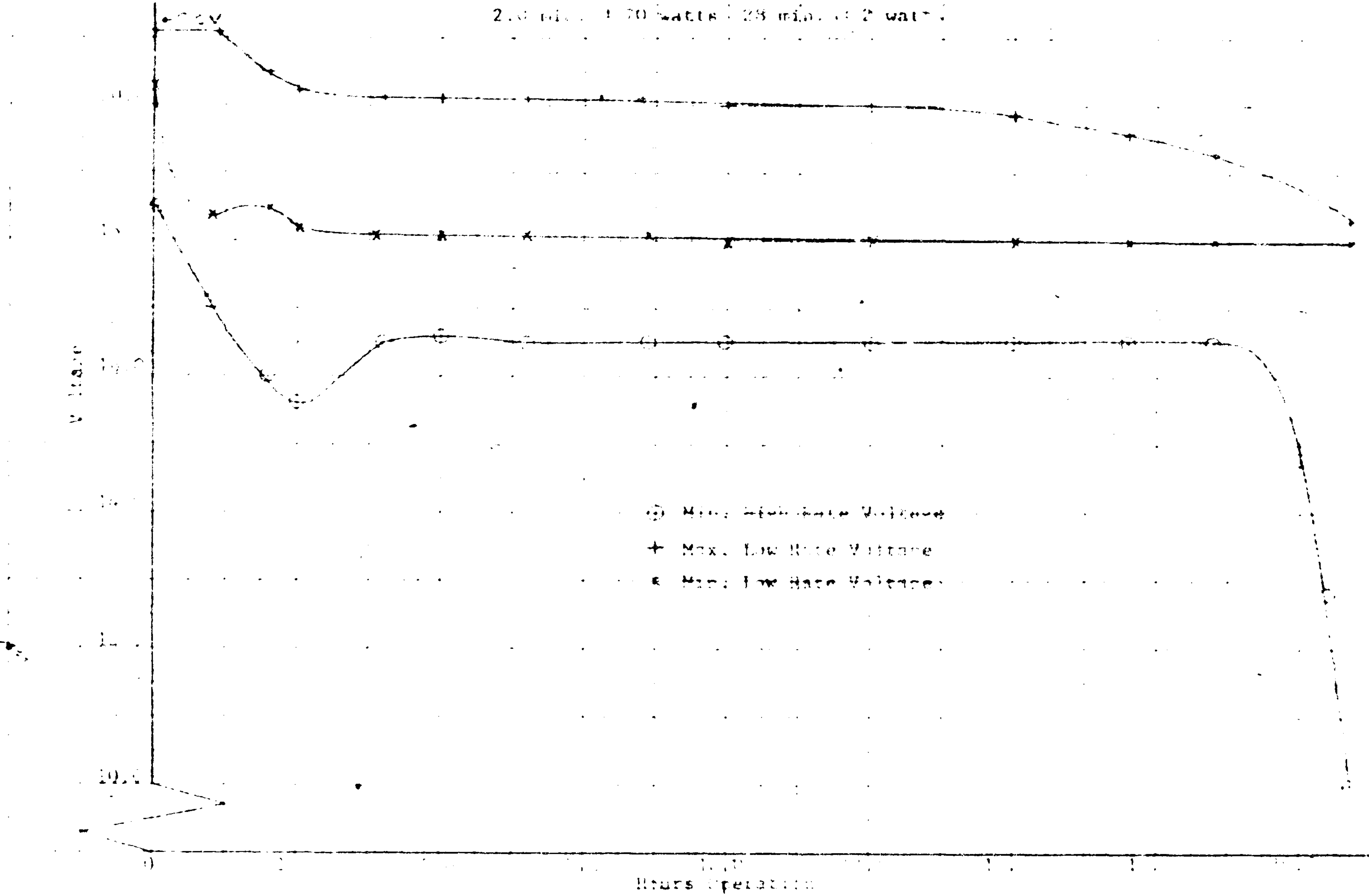
Two twelve cell units were constructed and tested in an attempt to attain a higher discharge voltage and to determine the optimum electrolyte concentration for -40°F discharge. One battery, S/N MAP-2014-3, was constructed with a sufficient quantity of magnesium perchlorate to yield a 3.0 normal electrolyte upon activation. The other unit, S/N MAR-2014-4, was constructed with a sufficient amount of magnesium perchlorate to yield a 2.0 normal solution.

These two batteries were conditioned for 16 hours at -40°F and activated with +40°F water. The units were discharged in series at the AN/PRC-62 specification rates. A polystyrene case as above was used to insulate these batteries during discharge.

As displayed by Figure Nos. 16 and 17, the units yielded lower average voltage per cell than the previous battery, MAP-4012-2, which was discharged at -40°F. Battery No. MAP-2014-3 utilizing 3.0 normal electrolyte yielded 8.7 ampere-hours capacity, or 91.5% positive efficiency after 19.5 hours of operation, while MAP-2014-4 containing 2.0 normal electrolyte yielded 8.46 ampere-hours capacity or

13

11-cell Battery, — F
Discharge Rate
2.0 min. 70 watts 28 min. 2 watts



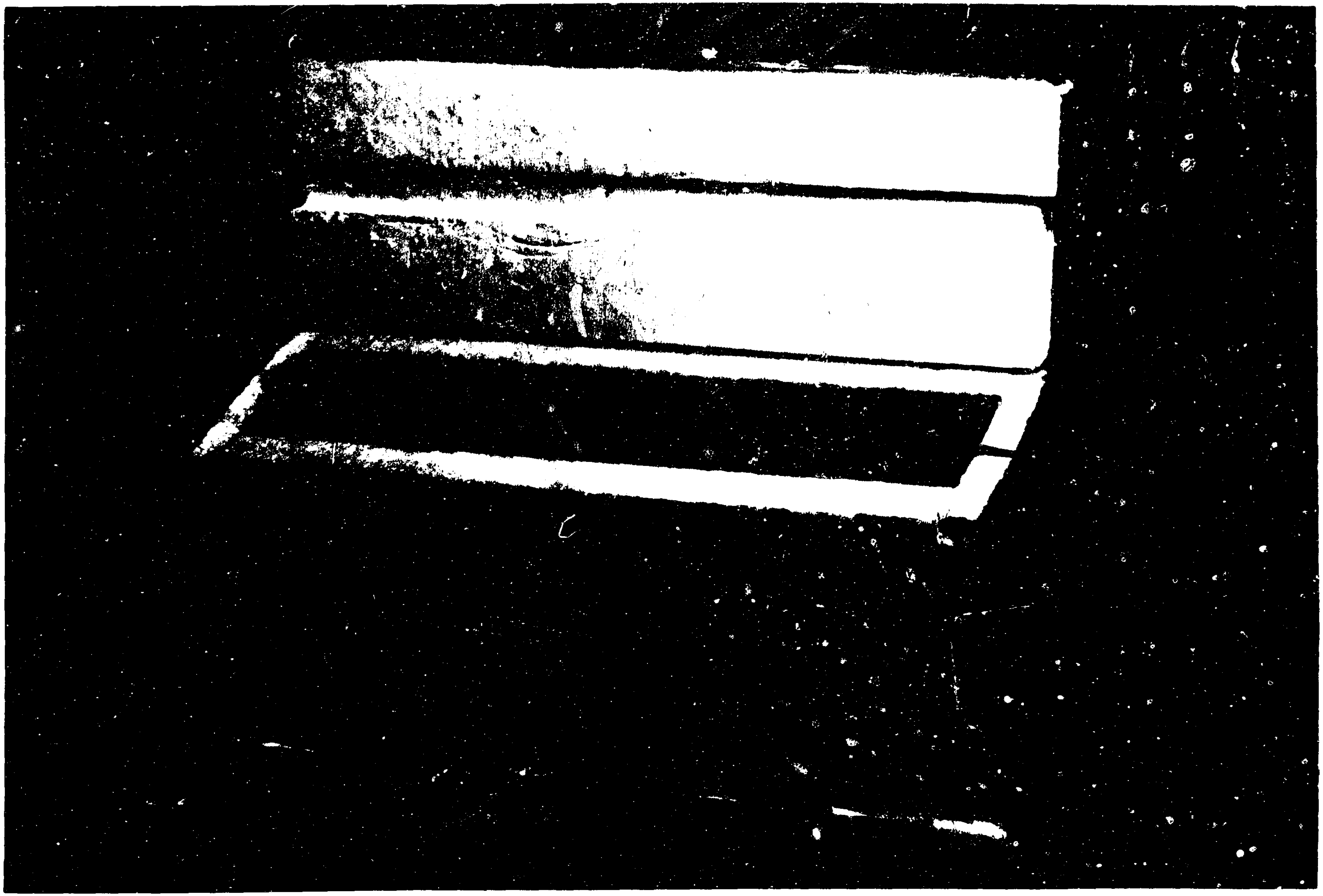


FIGURE NO. 14
MAGNESIUM/MAGNESIUM PERCHLORATE/MERCURIC OXIDE
BATTERY BEFORE DISCHARGE (INSULATED)
9.1 ampere-hours 91.4% efficient

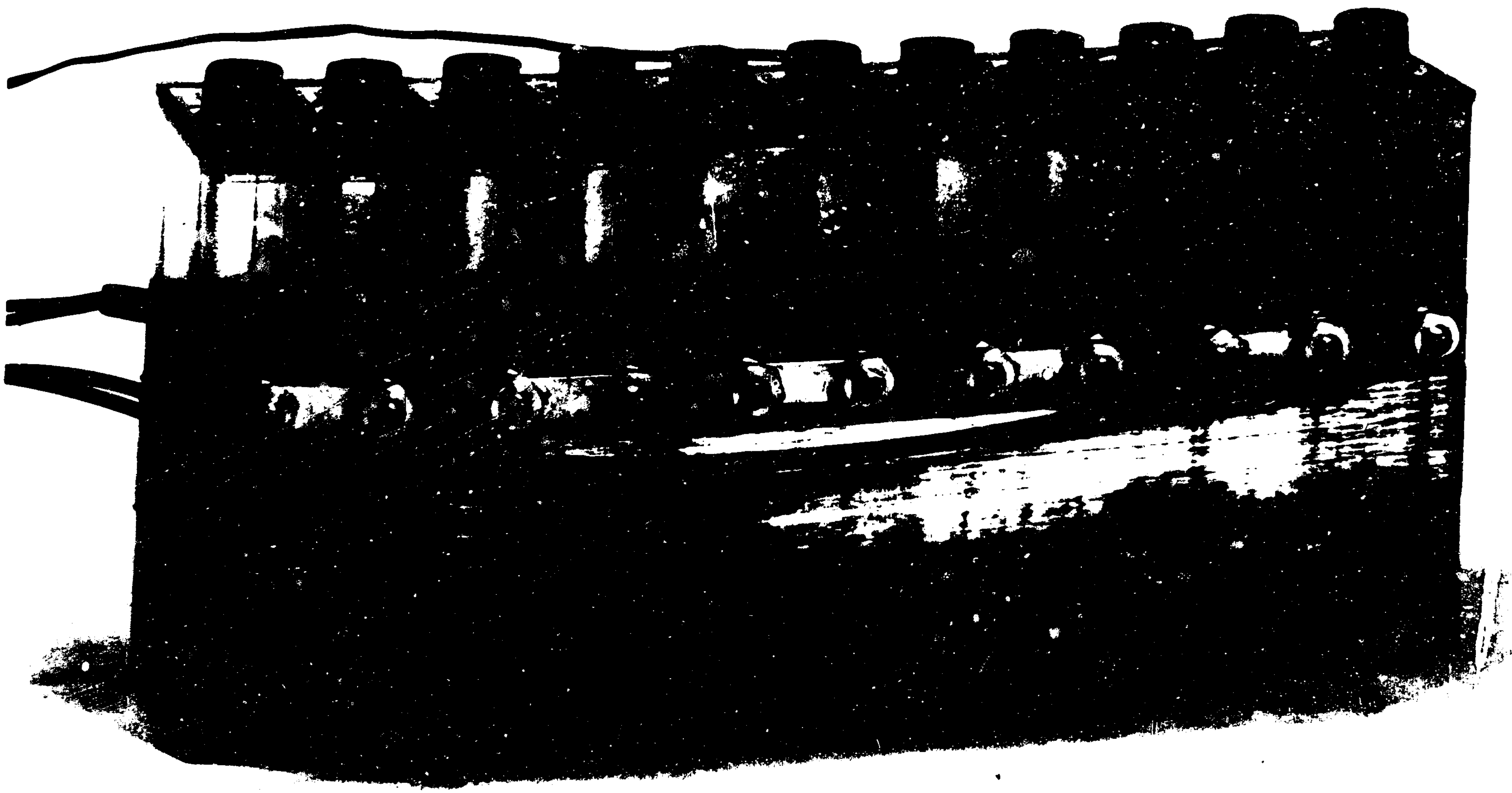
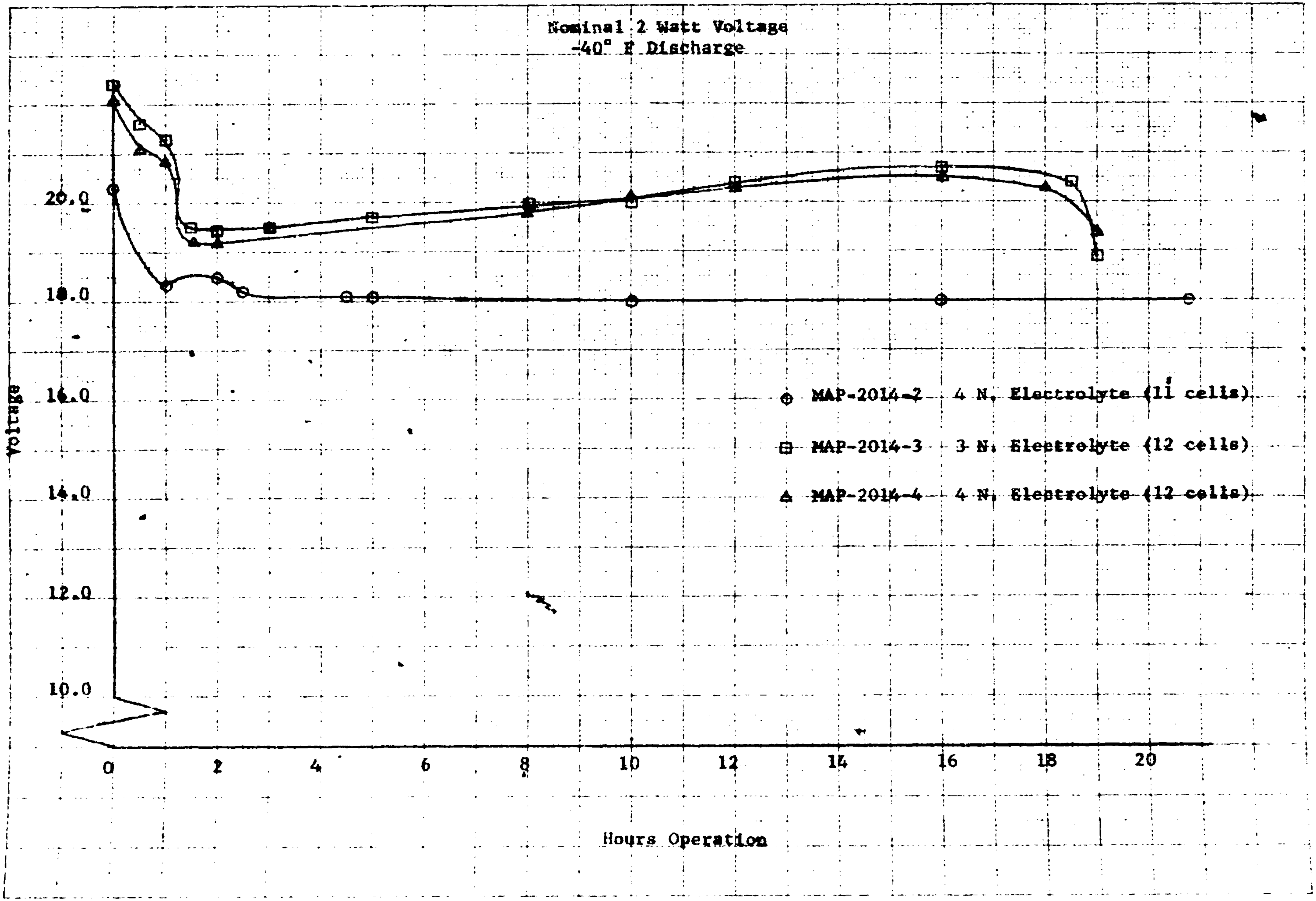


FIGURE NO. 15
MAGNESIUM/MAGNESIUM PERCHLORATE/MERCURIC OXIDE
BATTERY AFTER DISCHARGE (UNINSULATED)
9.1 ampere-hours 91.4% efficient

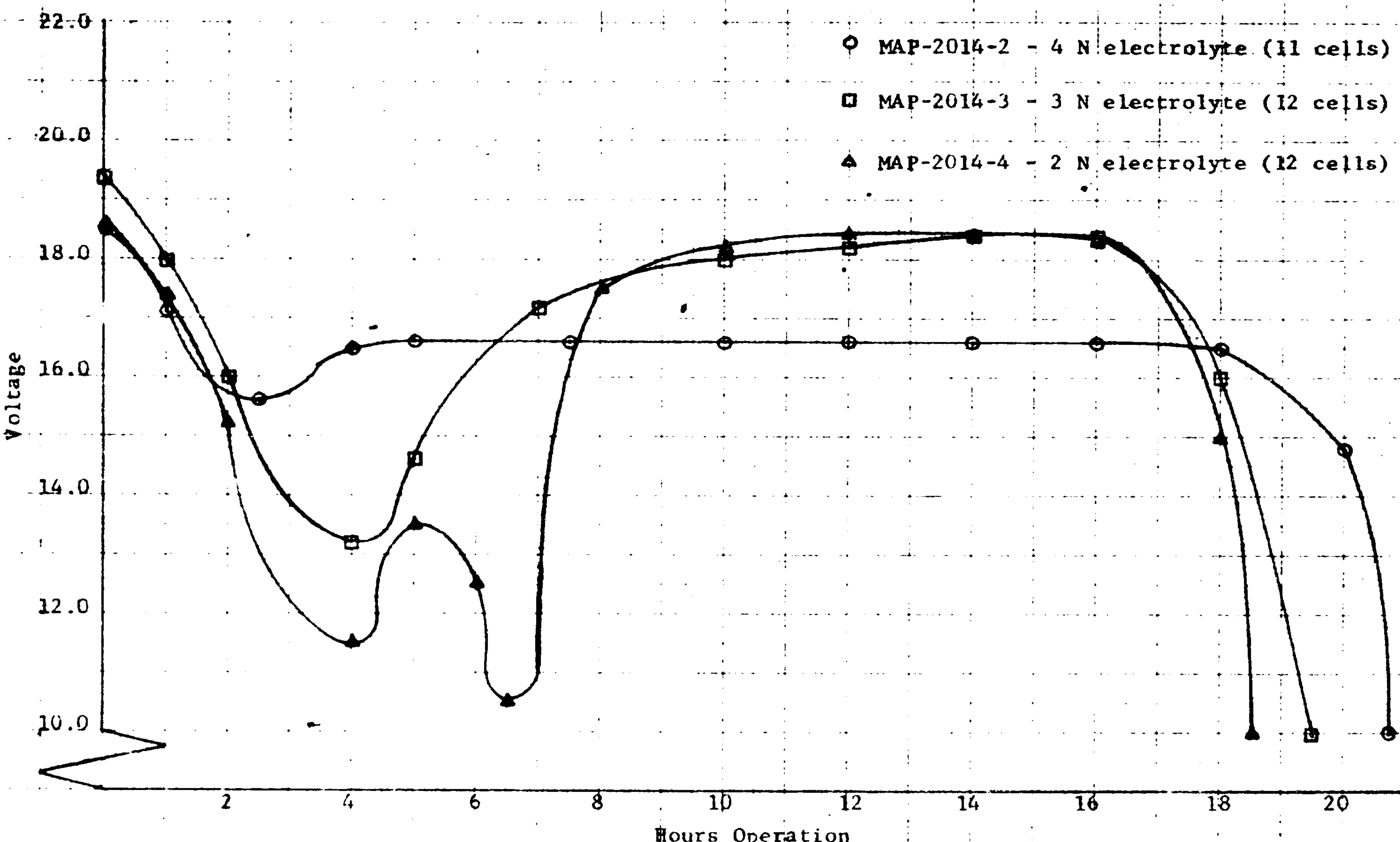
FIGURE NO. 16



Nominal 2 Watt Voltage
-40° F Discharge
Voltage
20.0
18.0
16.0
14.0
12.0
10.0
0
2
4
6
8
10
12
14
16
18
20
Hours Operation
MAP-2014-2 4 N. Electrolyte (11 cells)
MAP-2014-3 3 N. Electrolyte (12 cells)
MAP-2014-4 4 N. Electrolyte (12 cells)

FIGURE NO. 17

Minimum 70 watt Voltage
-40° F Discharge



89% positive efficiency after 18.5 hours operation. Both units displayed approximately 42 watt-hours per pound. The battery voltage of MAP-2014-4 declined to 10.5 volts during the high rate surge after 6.5 hours of operation. The battery voltage of MAP-2014-3, utilizing 3.0 normal electrolyte, decayed to 13.2 volts after three hours of operation. This indicates the minimum electrolyte concentration for -40°F discharge is approximately a 3.0 normal solution. Figure Nos. 18, 19 and 20 display the batteries prior to and after discharge.

3. Ten Cell Battery - (-40°F Discharge)

By comparing the discharge voltages and battery efficiencies at -40°F discharge, it appeared that the most satisfactory battery performance would be attained with a ten-cell unit utilizing a 4.0 normal electrolyte solution.

One ten-cell unit, MAP-2014, S/N 5, was constructed and conditioned as previous at -40°F for 16 hours. This battery was activated with +40°F water and discharged to the AN/PRC-62 specification rates. During the discharge the battery was insulated with polystyrene as previous. As shown in Figure No. 23, this battery displayed a more stable voltage than the twelve cell units, yielding 8.75 ampere-hours capacity, or 90.8% positive efficiency. The 43 watt-hours per pound displayed by this battery, or 1.67 watt-hours per cubic inch, was the highest attained to date at -40°F discharge. Construction of this unit was identical to the MAP-2014-6 displayed by Figure No. 21. This unit weighed 3.2 pounds activated. The physical dimensions were as follows:

Width (inches)	2.0
Length (inches)	8.87
Height	4.62

4. Ten-Cell Battery (+78°F Discharge)

One ten-cell battery, MAP-2014-6, was constructed identically to MAP-2014-5 which was discharged at -40°F. This battery was discharged at the AN/PRC-62 specification rates. As noted by Figure Nos. 22 and 23, this unit displayed voltage

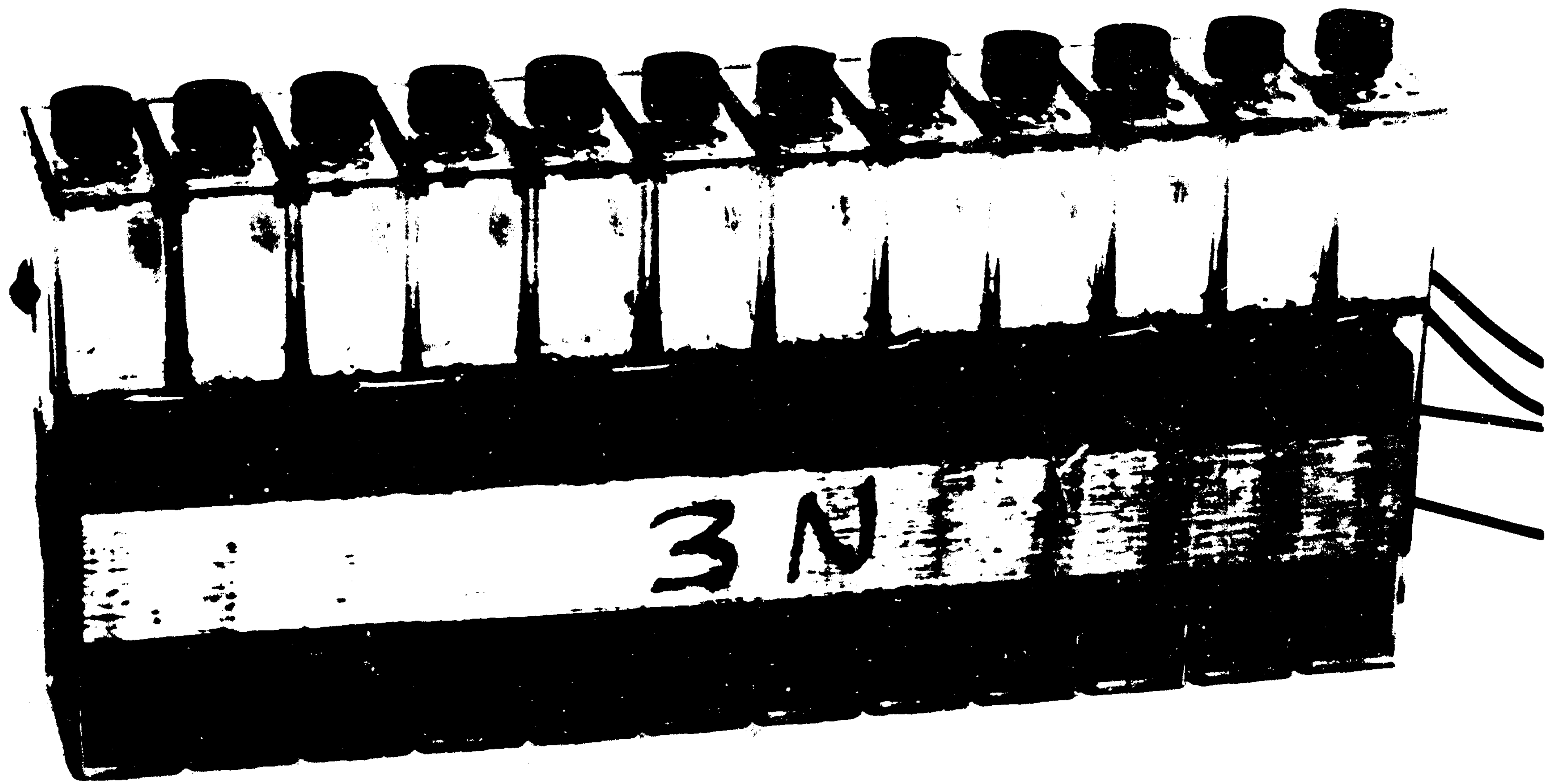


FIGURE NO. 18
MAP-2014-3 PRIOR TO ACTIVATION
3 NORMAL ELECTROLYTE

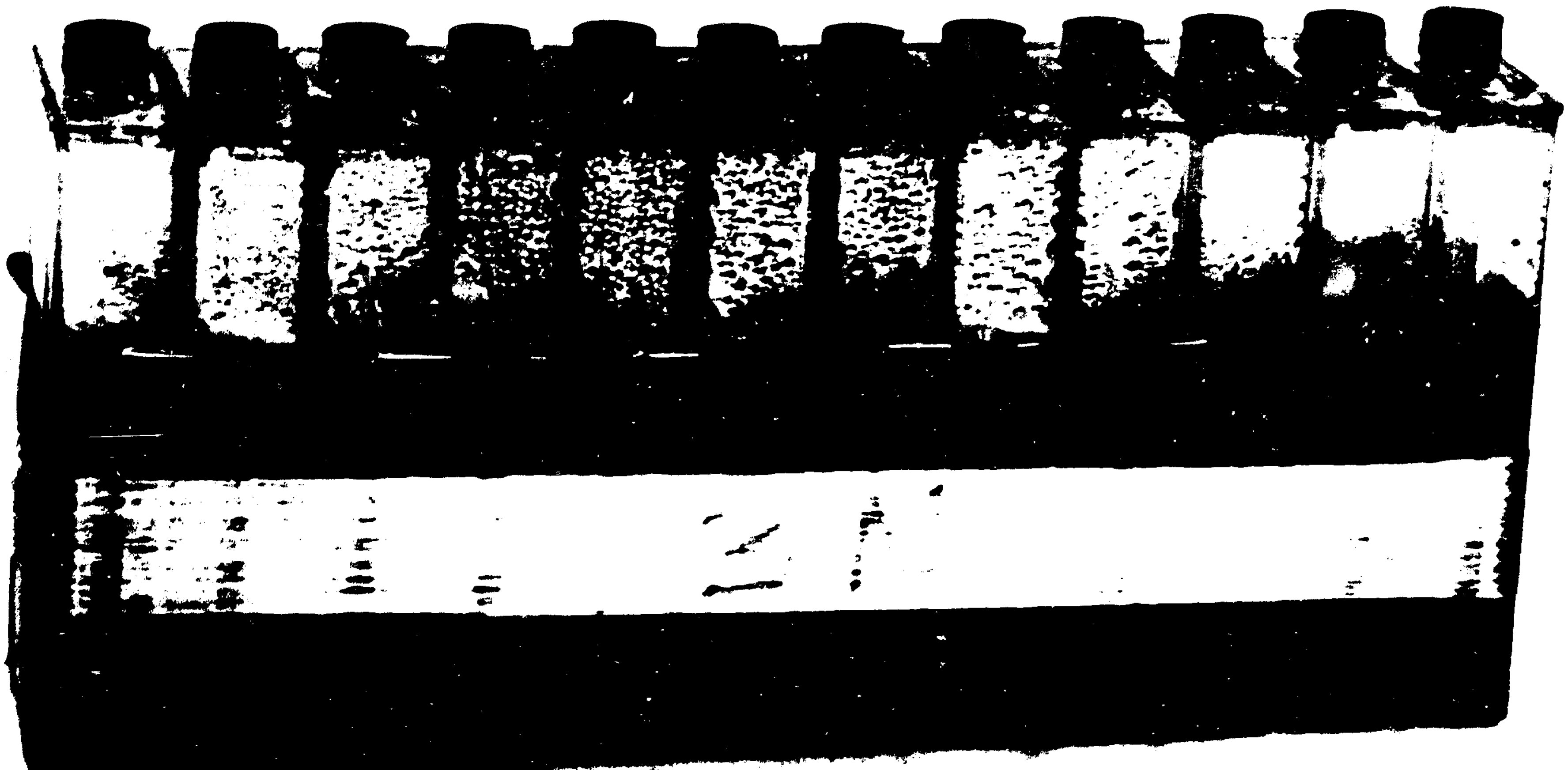


FIGURE NO. 19
MAP-2014-3 AFTER DISCHARGE
3 NORMAL ELECTROLYTE

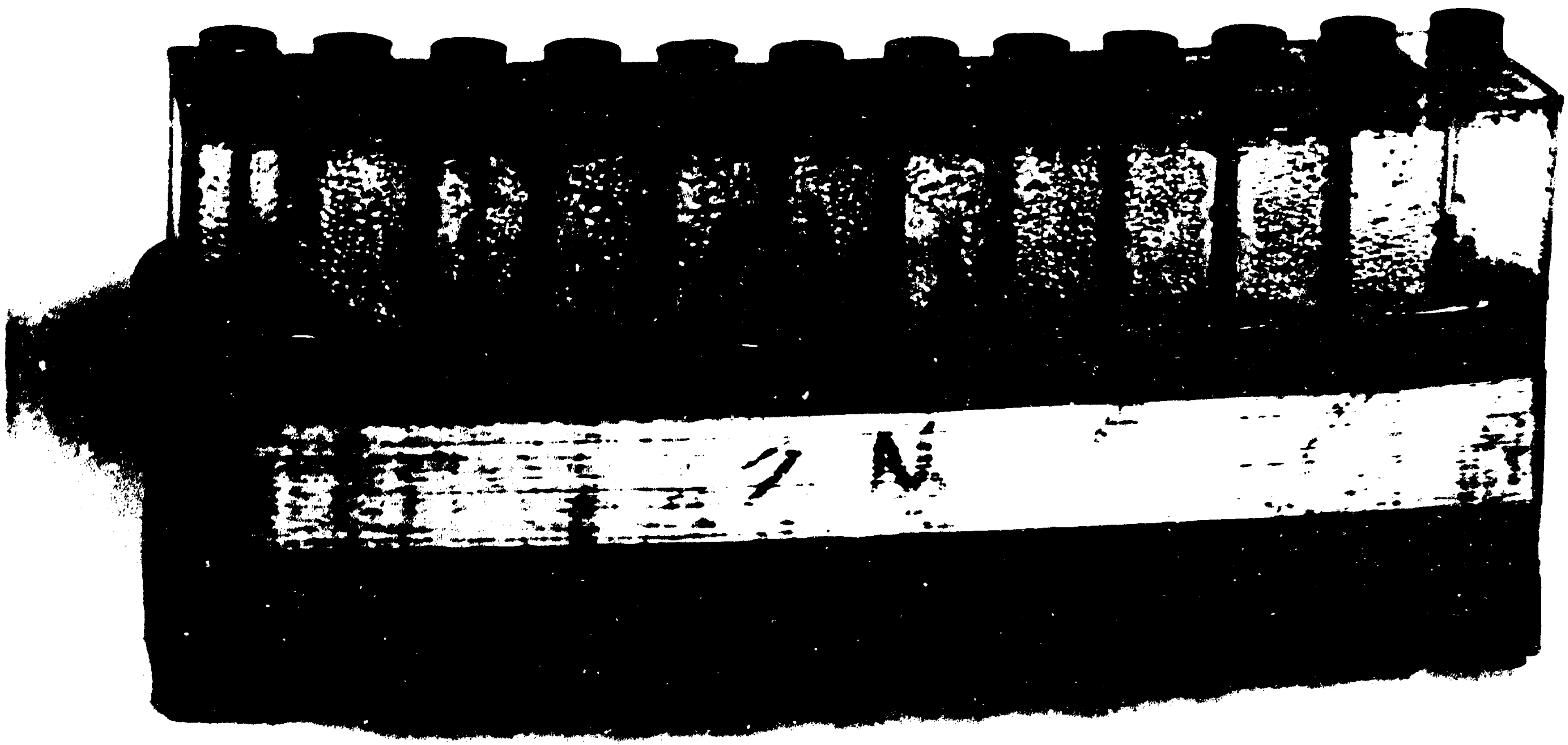


FIGURE NO. 20
MAP-2014-4 AFTER DISCHARGE
2 NORMAL ELECTROLYTE

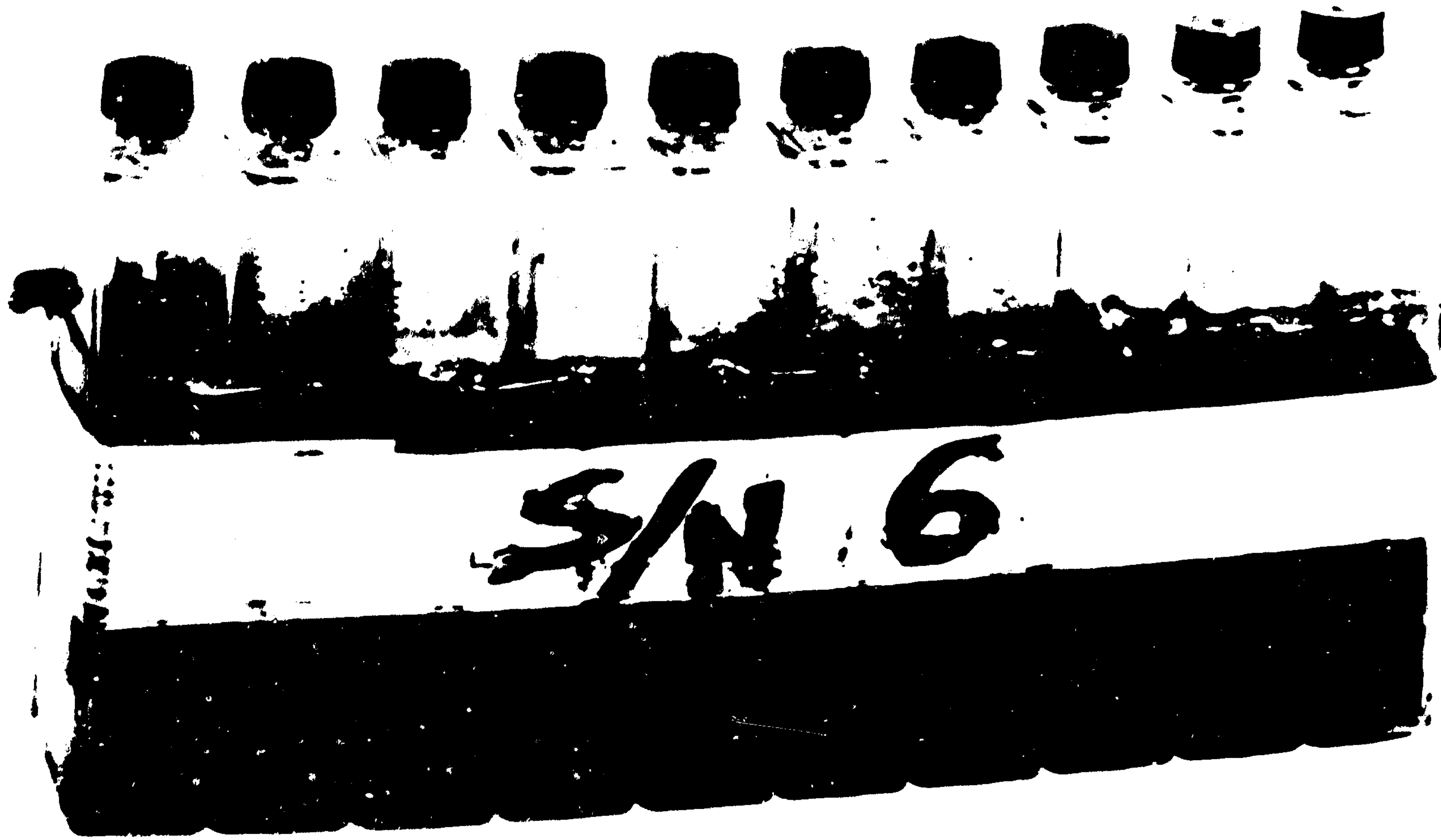


FIGURE NO. 21
MAP-2014-6 AFTER DISCHARGE
4 NORMAL ELECTROLYTE

FIGURE NO. 22

Nominal 2 Watt Voltage
(1[illegible] Cell Batteries)

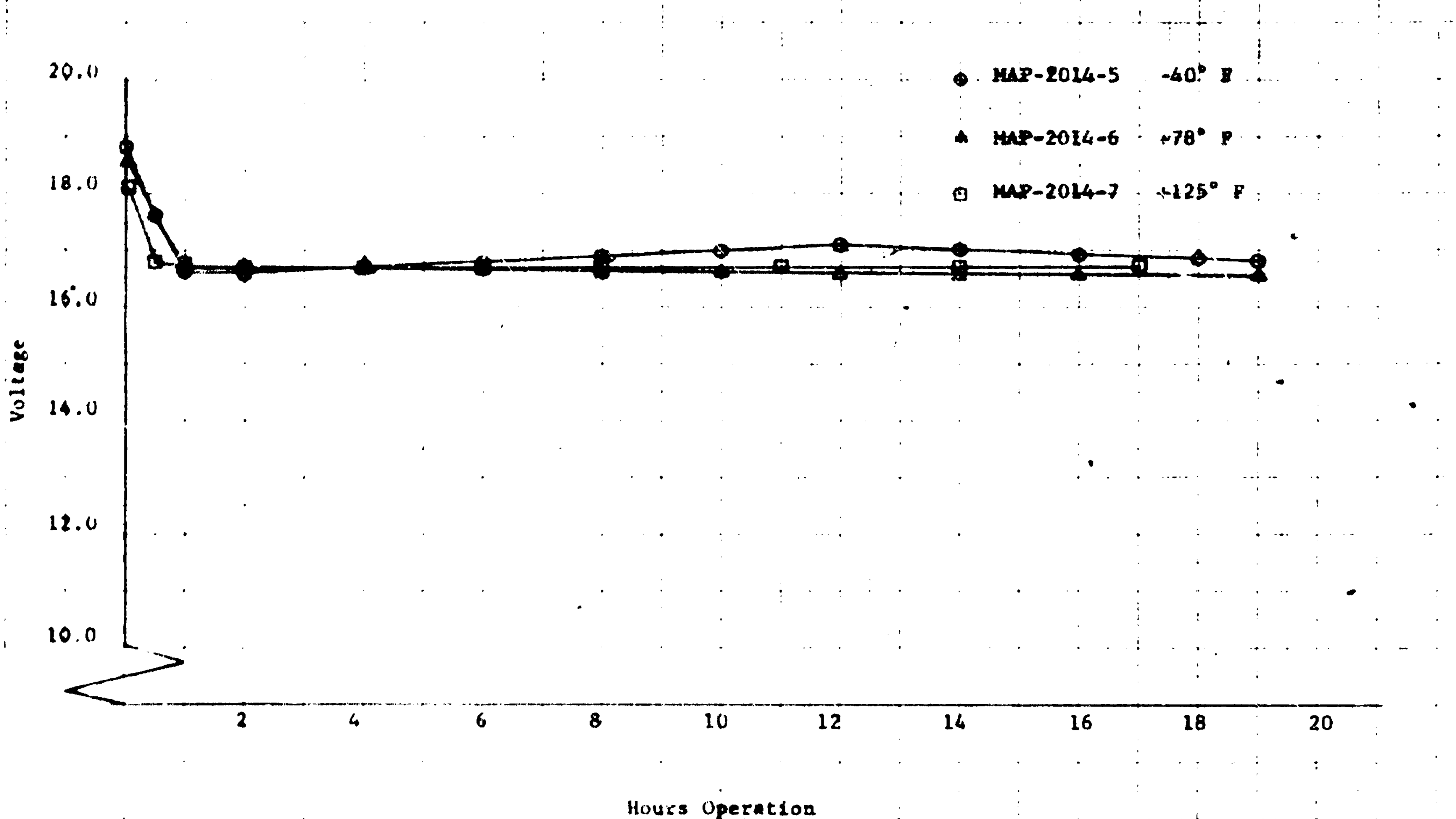
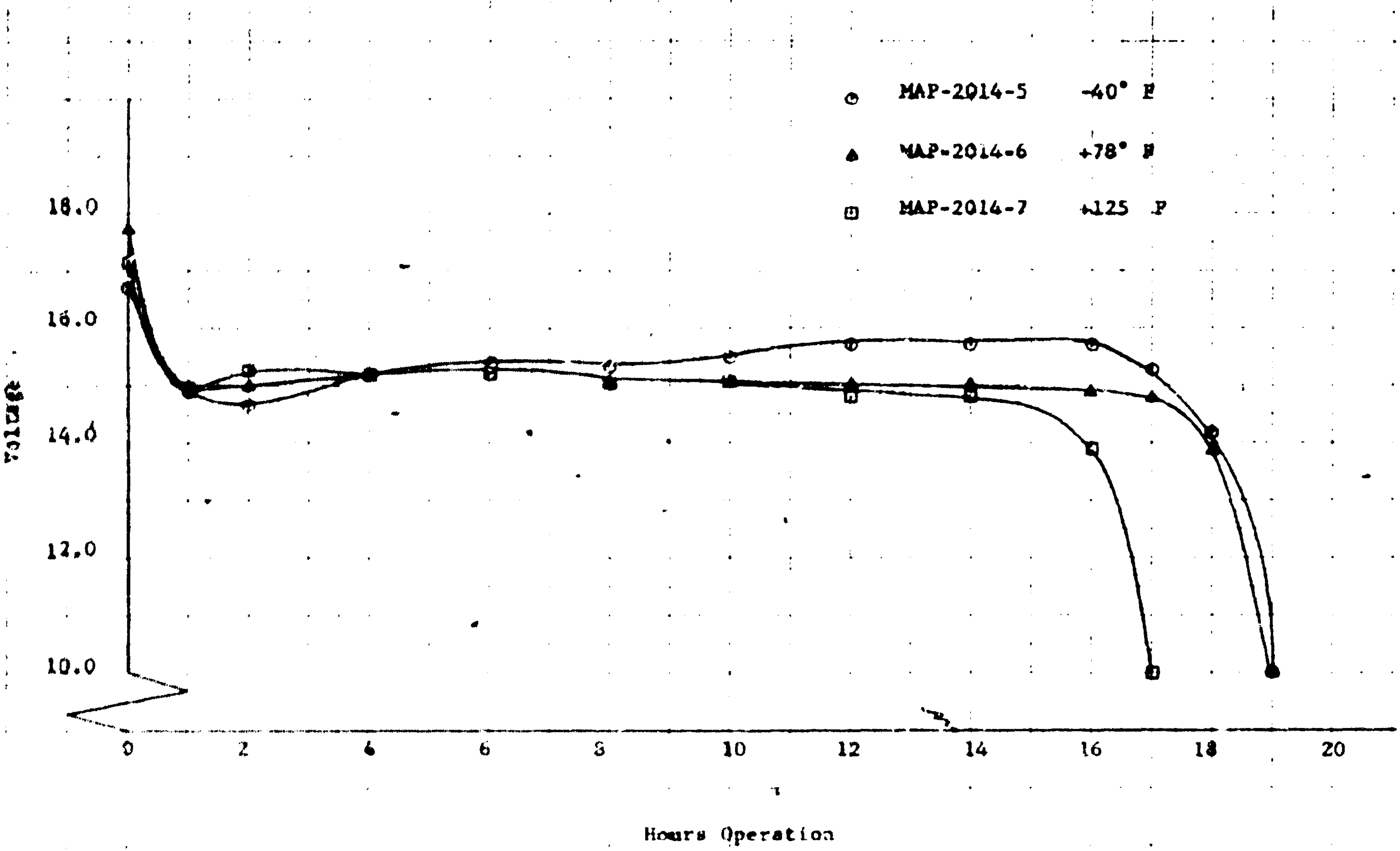


FIGURE NO. 23

Minimum 70 Watt Voltage
(10 Cell Batteries)



and capacity very similar to the -40°F ten-cell battery when discharged at +78°F. The battery was removed from cycle when the voltage declined to 10.0 volts at the high rate surge after 19 hours operation. This battery yielded 8.45 ampere-hours, or 41.7 watt-hours per pound. A monitor of the battery temperature indicated the maximum temperature was +148°F. This occurred immediately upon activation while the minimum temperature of +95°F occurred approximately 2.5 hours after activation. Nominal operating temperature was approximately +105°F.

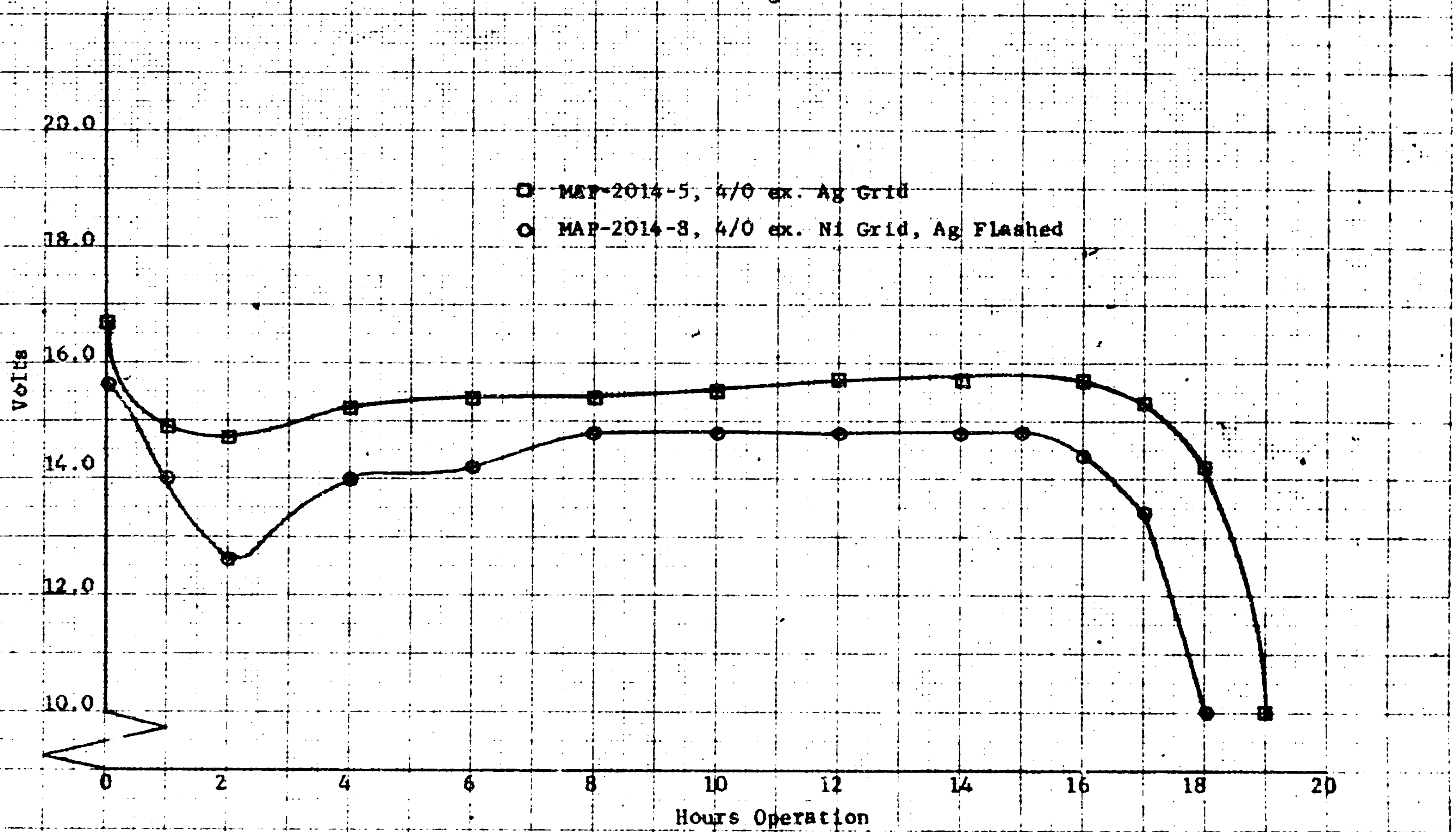
5. Ten Cell Battery - (+125°F Discharge)

A ten-cell battery, MAP-2014-7, was constructed identical to MAP-2014-5 and 6. This battery was conditioned at +125°F for 16 hours and activated with water stored at that temperature. To minimize the heat of solution of the magnesium perchlorate and still maintain a sufficiently concentrated electrolyte for battery discharge, each cell of this unit was activated with 35 ml of water instead of 25 ml normally used. This yielded a 2.86 normal electrolyte concentration. A monitor of the battery temperature throughout the discharge indicated the maximum temperature of +174°F occurred immediately upon activation while the minimum temperature of +132°F occurred approximately 2.5 hours after activation. Nominal battery operating temperature was approximately +150°F. As displayed by Figure No. 23, the battery voltage was approximately the same as that displayed by batteries discharged at +78°F and -40°F. However, the capacity was less. This was attributed to a decrease in cathode efficiency at the higher temperature. The unit yielded 7.68 ampere-hours after 17 hours of operation or 35.2 watt-hours per pound. A comparison of the discharge voltages of the -40°F to +125°F discharges is displayed by Figure Nos. 22 and 23.

6. Ten-Cell Battery - (-40°F Discharge)

A ten-cell battery, MAP 2014-8, was constructed utilizing 4/0 expanded nickel grid Ag plated. Figure No. 24 shows where MAP-2014-8 discharges at a lower

FIGURE NO. 24
MINIMUM 70 WATT VOLTAGE
(10 cell batteries)
-40° F Discharge



voltage as compared to MAP-2014-5. However, the nickel grid battery remains within specification and is the modular battery for the AN/PRC-62 application.

G. Initiation of the PPS-5 Application

The PPS-5 application was studied and the initial design study was to build a multiplate type construction of the Eagle-Picher Y-12 "A" cell.

Dimensions of the Eagle-Picher Y-12 are:

Height	6.875 inches
Width	5.234 inches
Length	1.437 inches
Volume, cu. in	51.7 in^3

This Y-12 construction is modified to fit the application. The element design was as follows:

Number plates/cell, pos./neg	18/19
Plate size, height/width, in.	4.25 x 5.0
Pos. plate thickness, in.	.032
Neg. plate thickness, in.	.015
Type Grid P/N	.4/0 ex. Ag
Container, type	EP Y-12, modified
Inside height, in.	4.625, 1.250 electrolyte reservoir
Inside width, in.	5.23
Inside length, in.	1.437
Pos. material, gm/sq. in. (including grid)	1.75 88% HgO
Pos. formulation:	
HgO	92%
CMC	1.5%
Carbon	6.0%
Fiber	0.5%
Neg. material, gm/sq. in.	0.41 gm/in^2
Pos. material, gm/cell	670 gm.
Neg. material, gm/cell	166 gm.
Theoretical capacity, pos/neg A.H.	146/365
Pos. material density, gm/cu. in.	59
Neg. material density, gm/cu. in.	28.6
CC of H_2O per cell	260 + 116 gm. $Mg(ClO_4)$, 2.6 gm. Li_2CrO_4
Total cell weight	3.54 lb activate
Expected capacity, A.H.	120 A.H. @ 10 amp. to 10% voltage drop

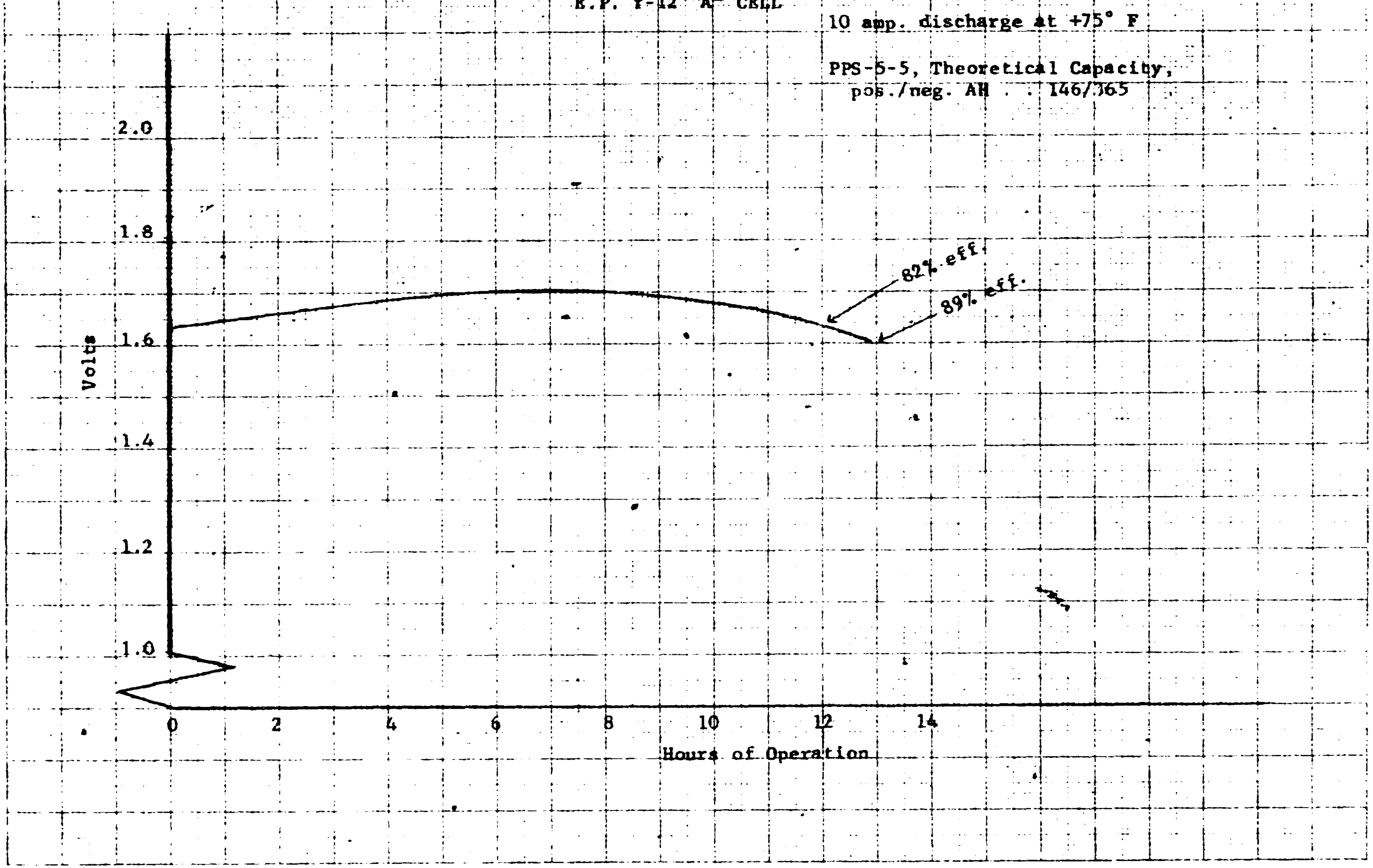
This cell ran at +75°F for 13 hours at 10 amps with a nominal voltage of 1.67 (Figure No. 25). It was felt that this was a good initial start and the development effort will be carried over into the next quarter.

The estimated weight and volume required to meet the PPS-5 application are as follows:

Four (4) cells at 3.6 lb/cell	14.4 lbs.
Height, in.	6.0
Width, In.	5.4
Length, in.	6.5
Volume, cu. in.	210.2
watt-hours/lb	60

FIGURE NO. 25

PPS-5
E.P. Y-12 "A" CELL



VI. SUMMARY

The progress that has been made during this quarter signifies the potential of the magnesium/magnesium perchlorate system. A reliable magnesium/magnesium perchlorate battery has been developed to meet the AN/PRC-62 application. A large advancement was made with the initial launch of the PPS-5 application.

Concentrating on the optimum performance of the PPS-5, the following phases should have further investigation:

(1) Battery assembly for the PPS-5;

(2) Battery container for the AN/PRC-62 and PPS-5 for final production;

(3) Continued research on the PPS-5 with emphasis on watt-hours per pound.

VII. CONCLUSIONS

Research data has indicated that production cells and batteries can be built which perform within the AN/PRC-62 requirement in the temperature range of -40°F to +125°F. These batteries will perform within relatively close voltage limits at temperatures from -65°F to +125°F when the discharge load is continuous.

Initial study indicates that cells and batteries can be produced to meet the PPS-5 application. Efforts for the next quarter have been centered around developing a battery which will meet the PPS-5 specifications.

VIII. PROGRAM FOR NEXT INTERVAL

Cell and battery design studies:

a). Cathode blends and mixes,

b). Grid study for PPS-5 application,

c). Battery design for the PPS-5 application,

d). Final assembly of the AN/PRC-62,

e). Development of the AN/PRC-25

The program for the next interval will include the phases outlined. The general program will be directed toward meeting the requirements of the PPS-5 application.

IX. PERSONNEL

NAME	TITLE		MAN-HOURS
Cupp, E. B.	Project Engineer		273
Sharpe, J. R.	Design Engineer		512
Morse, E. M.	Engineering Manager		37
Dines, J. M.	Senior Engineer		21
McCleary, E.	Battery Engineer		215
Gosch, C. O.	Staff Engineer		10
Hodges, K.	Technician		522
		TOTAL	1,590

A P P E N D I X